Temperature Capability for *In-Situ* TEM Nanostage

Mary LeBlanc
(925) 422-8954
leblanc2@llnl.gov

Nanomaterials are roughly defined as solids with characteristic dimensions that are 200 nm or less. The physical properties of nanomaterials are frequently very different from typical bulk properties. For example, some newly invented composite materials that incorporate nanomaterials in their structure have very high yield strength and excellent fracture toughness. In essence, it is the very small dimensions and concurrent high surface-area-to-volume ratio of the nanomaterials that give rise to these properties. It is important to be able to characterize the mechanical behavior of these materials under different temperature regimes, especially as nanomaterials and structures are used in various programmatic applications including laser targets, materials for weapons, and sensing elements.

Perhaps the most compelling application for the Transmission Electron Microscope (TEM) nanostage is to directly measure dislocation velocities, which are important material characteristics used in multiscale modeling. The nanostage also permits the direct observation of dislocation reactions and the resulting dislocation substructures, which can be used to validate dislocation dynamics simulations.

Project Goals

The goal of this project is to establish a new capability to experimentally measure the mechanical response of nanomaterials and structures in the TEM, over a range of temperature from 100 to 500 K. To accomplish this goal, a loading stage must be built that is compatible with the TEM, and a loading cartridge, which holds the nano-sized sample, must be fabricated to fit into the tip of the stage and transfer a continuously measured load onto the sample (see Fig. 1). The scope of the project is to establish and test the new capability, and provide documentation on the utility for future studies.

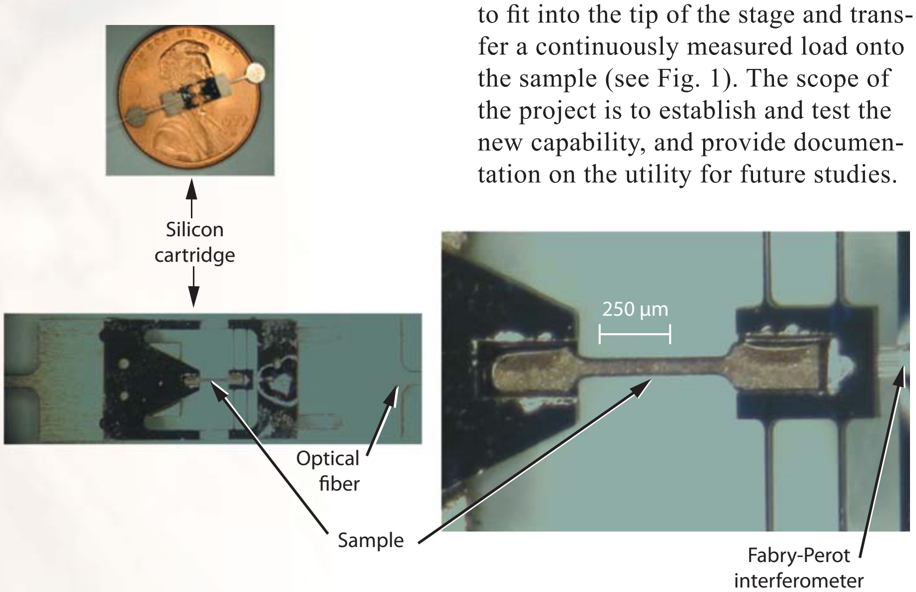

Figure 1. Photographs of the loading cartridge. Counterclockwise: the cartridge in relation to a penny; the complete cartridge; blow-up near the sample region. The sample has not yet been thinned to transparency.

Relevance to LLNL Mission

This project will add to the increasing number of capabilities LLNL will need to characterize and use nanomaterials and structures. Nanomaterials have the potential to play a key role in the development of sensors for programmatic applications.

The first use of this capability will be to quantify the dislocation velocity as a function of applied stress in single crystals. These dislocation mobility values have never been accurately measured, and are essential input for LLNL's multiscale modeling program.

FY2006 Accomplishments and Results

Sample Fabrication. Damage to the test sample during handling of the cartridge was an unexpected and significant obstacle during FY2006. In response, fixturing to hold the loading cartridge during Focused Ion Beam thinning of the sample was fabricated. The titanium frame for the cartridge was stiffened to reduce in-plane and out-of-plane bending, and an aluminum reinforcing plate was bonded to the cartridge to prevent damage during installation into the straining stage. Successful thinning of the test sample to electron transparency has since been demonstrated by our collaborator at LBL (Fig. 2).

Loading stage. The *in-situ* TEM straining stage has been fabricated, assembled, and tested (Fig. 3). It is sized to fit in LLNL's Philips 300 TEM. The tip of the stage is cooled by a cold finger that resides inside the microscope and is attached after the stage is inserted. Temperature measurements made in a vacuum chamber to simulate the microscope environment (Fig. 4) show that the loading cartridge temperature matches that of the tip.

Data acquisition. A PC-based data acquisition system has been assembled, and software written to capture the TEM image (either from the microscope camera or an external camera), and up to eight channels of other data, including load and temperature.

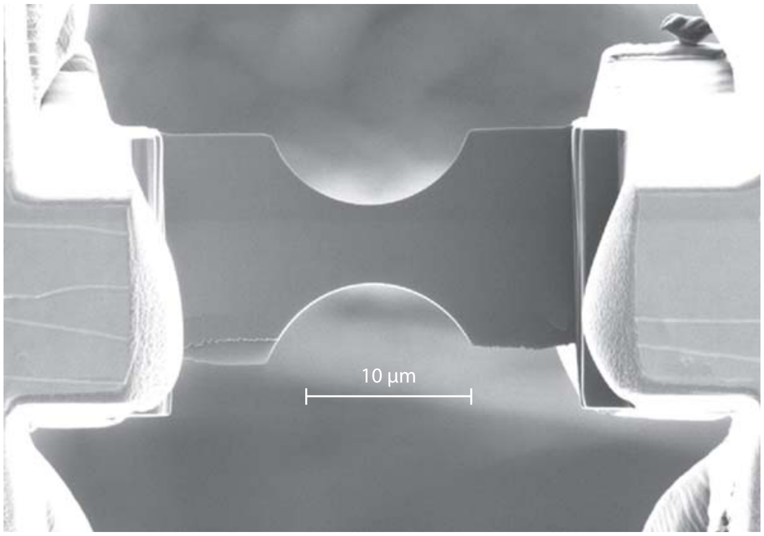

Figure 2. SEM photograph of the sample thinned to electron transparency. The cross-section is diamond shaped with the minimum thickness (1.6 µm) near the sample center.

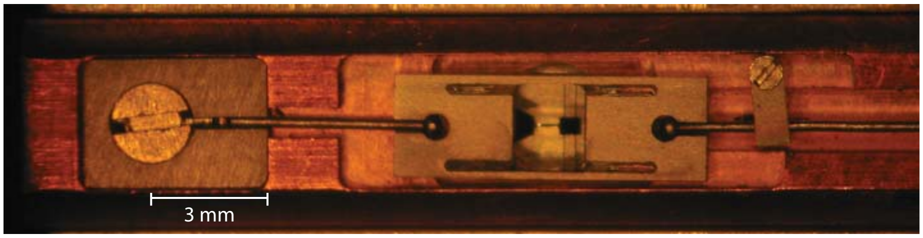

Figure 3. Close-up of the stage tip with a loading cartridge installed. The cartridge is in unloaded condition (loading wires slack) with the silicon side down.

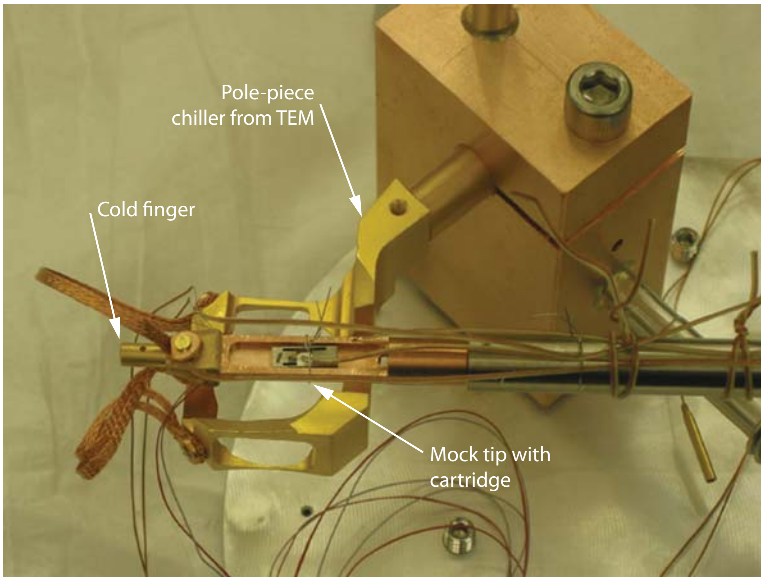

Figure 4. Photograph of the temperature conduction experiment. A mock tip was cooled using conduction from the LN-cooled pole-piece chiller through copper braid to the cold finger that attaches to the stage tip. The cartridge and tip were within 1 °C; the cold finger was 5° cooler.